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Final Report

SURFACE, INTERFACE, AND BULK PROPERTIES OF HIGH T<sub>c</sub>  
SUPERCONDUCTORS

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## FINAL REPORT

*The Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8</sub> material*

We have performed extensive studies of the Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8</sub> material ever since its discovery in early 1988. Single crystals, polycrystalline pellets and thin film samples of the Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8</sub> material were examined using various photoemission related techniques. Especially the single crystalline Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8</sub> material was found to have an extraordinary inertness in ultrahigh vacuum conditions, allowing a detailed examination of the surface electronic structure. Consequently, most of our investigations were focused on the single crystalline materials. Low-Energy Electron Diffraction (LEED) was used to confirm the single crystallinity of the *in situ* cleaved crystals of Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8</sub>. Sharp diffraction spots indicative of long range periodicity were observed. The observed LEED pattern showed that the Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8</sub> material preferentially cleaves parallel to the a-b plane. In addition, a superstructure was seen extending along one of the symmetry axes in the a-b plane. The superstructure was found to have a periodicity about 5 times as long as the cell dimensions of the a and b axes, in agreement with the bulk structure. The LEED patterns were reproduced after several days in ultrahigh vacuum, showing the inertness of the single crystalline materials. Comparison of the spectral features of the single crystalline and polycrystalline samples of Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8</sub> was made, showing an overall good agreement. More specifically, for both types of samples an appreciable density of states at the Fermi level was observed, showing the metallic nature of these materials. Moreover, single component O1s emission, suggestive of contamination-free surfaces, and a Cu2p satellite to main line intensity ratio of 0.3-0.4 were found for both samples. The presence of a strong Cu2p satellite is taken as evidence that the Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8</sub> material contains unfilled 3d electrons. Studies of the Bi<sub>2</sub>Sr<sub>2</sub>CuO<sub>6</sub> and Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8</sub> materials showed that the density of states at the Fermi level is slightly lower in Bi<sub>2</sub>Sr<sub>2</sub>CuO<sub>6</sub> than in Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8</sub>. In addition, the states at the Fermi level appear to have less Cu character than those of the earlier

superconductors of  $\text{La}_{1.8}\text{Sr}_{0.2}\text{CuO}_4$  and  $\text{YBa}_2\text{Cu}_3\text{O}_7$ . Detailed studies of the core level photoemission spectra from  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  showed evidence of photoemission loss structures, that were assigned to plasmon excitations and one-electron intra-atomic transitions. By depositing Rb followed by exposure to molecular oxygen, we were able to extract fundamental information about the  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  materials. For both polycrystalline and single crystalline samples, deposition of Rb resulted in disrupted Bi-O bonds, and the formation of additional oxygen states at the surface. Rb-deposition on single crystals of  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  firmly shows that this material preferentially cleaves between adjacent Bi-O layers. Synchrotron radiation studies using both angle-resolved and angle-integrated analyzers were performed on single crystals of the 90 K superconductor  $\text{Bi}_2(\text{Sr,Ca,L})_2\text{Cu}_2\text{O}_8$ . The angle-resolved measurements showed essentially dispersionless character of the valence band states as a function of the wave vector component parallel to the c-axis, while clear dispersion of the order of 0.3-0.4 eV was observed as a function of the wave vector component parallel to the a-b plane. This result, which is consistent with the macroscopic properties of the  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  material, confirms that virtually all the hybridization among the Cu3d and O2p states is confined to the two-dimensional layers. Absorption measurements were performed on the  $\text{Bi}_2(\text{Sr,Ca,L})_2\text{Cu}_2\text{O}_8$ ,  $\text{BaBiO}_3$ , and  $(\text{Nd,Ce})_2\text{CuO}_4$  systems by recording spectra in the Constant-Final-State (CFS) mode. Absorption signals from all elements but Ce were identified. Comparison of the Bi absorption signals of  $\text{Bi}_2(\text{Sr,Ca,L})_2\text{Cu}_2\text{O}_8$ , and  $\text{BaBiO}_3$  shows that the Bi6s states are occupied differently in the two compounds, in agreement with predictions of band calculations. The intrinsic two-dimensionality of the  $\text{Bi}_2(\text{Sr,Ca,L})_2\text{Cu}_2\text{O}_8$  material was confirmed using polarization-dependent CFS spectroscopy. The results suggest that the electronic charge distribution is highly localized to the two dimensional layers. In addition, polarization-sensitive absorption signals due to Bi6s $\rightarrow$ 6p transitions show that the empty Bi6p<sub>z</sub> states are located at slightly higher energy than those of Bi6p<sub>x,y</sub> symmetry. As for the  $\text{La}_{1.8}\text{Sr}_{0.2}\text{CuO}_4$  and  $\text{YBa}_2\text{Cu}_3\text{O}_7$  materials, ion-bombardment of polycrystalline and thin film samples of the  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  material was found to adversely affect the electronic structure at the surface, as signaled by a rapid reduction of the copper valency to its monovalent state.



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### *Studies of interfaces between metals and $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$*

We have studied interfaces of the  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  superconductor with various metals using ultraviolet and x-ray photoemission spectroscopy, aiming at gaining insight of both scientific and practical nature. Perturbing the surface of the superconductors by depositing thin overlayers and monitoring the chemical and electronic structure changes that occur are a useful method for probing the properties of the superconductor itself. Also, electronic applications of superconductors will depend heavily on interface properties.

To study the effects of reactive metallic overlayers, deposition of Al and Rb onto single crystals of  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  was performed, and the effects of the two adsorbates were compared. Al shows a strong chemical reaction with all of the layers in the  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  compound. Al diffuses into the  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  substrate and forms bonds preferentially with the oxygen ions, and thus disrupts the chemical structure of the superconductor. Rb on the other hand, shows a reaction with only the Bi and O ions at the surface layer, presumably because its electropositivity and large ionic radius prevent it from penetrating into the bulk of the substrate. The results hence show that while Al adsorbates adversely affects the electronic structure of all layers of the  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  compound, the effects of Rb is primarily confined to the first atomic layer.

We have also studied overlayers of the noble metals Ag and Au on substrates of single crystalline  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ . Both of these metals are generally non-reactive and might be expected to form non-disruptive interfaces. Ag, however, appears to diffuse into the  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  material and shows a small chemical reaction. The important Cu valency is reduced in the surface region when a Ag overlayer is deposited. Au, on the other hand, seems to form a non-reactive interface with the  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  substrate, and thus shows promise as a means of passivating the  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  surface. No sign of significant reaction with the substrate can be detected, and the Au overlayer also seems to grow in a layer by layer fashion without forming islands or diffusing into the  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  substrate. Overall, Au appears to be the most suitable

material for making metallic contacts with the  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  material without altering the electronic structure of the substrate.

*The  $\text{Y}_{1-x}\text{Pr}_x\text{Ba}_2\text{Cu}_3\text{O}_{6+d}$  and  $\text{Pb}_2\text{Sr}_2\text{PrCu}_3\text{O}_{8+d}$  systems*

An other example of how we have used spectroscopic methods to gain some insight into the electronic structure and its relationship to the other properties of the high temperature superconductors is our group of studies of the quenching of the superconductivity in the  $\text{Y}_{1-x}\text{Pr}_x\text{Ba}_2\text{Cu}_3\text{O}_{6+d}$  (123 compound) and  $\text{Pb}_2\text{Sr}_2\text{PrCu}_3\text{O}_{8+d}$  systems. It is found that the superconductivity in the  $\text{Y}_{1-x}\text{Pr}_x\text{Ba}_2\text{Cu}_3\text{O}_{6+d}$  system is quenched as Y is replaced by Pr. This result is unusual because most of the  $\text{RBa}_2\text{Cu}_3\text{O}_{6+d}$  compounds, where R is a rare earth element, are superconducting with  $T_c$  near 90 K except for the cases of Ce, Pr and Tb (for Ce and Tb the 123 compounds do not form). A model for the  $T_c$  quenching could be that the valence of Pr is 4+ instead of 3+ as in the other rare earths. The extra charge would presumably be transferred to the  $\text{CuO}_2$  planes, filling the holes that are widely believed to be the superconducting carriers. We have performed photoemission, resonant photoemission, and Bremsstrahlung isochromat spectroscopy (BIS) on these materials. We found Pr to have a valency of 3+, instead of the proposed 4+. Our data points to another mechanism for the quenching of the superconductivity based on the strong Pr 4f / O 2p / Cu 3d hybridization which we have observed. This hybridization would alter the electronic or the magnetic structure of the  $x=0$  material, resulting in the quenching of the superconductivity. This result is consistent with conclusions drawn from the pressure dependence of the transport properties of these alloys.

As a further test, we performed similar measurements on the  $\text{Pb}_2\text{Sr}_2(\text{R}=\text{Pr})\text{Cu}_3\text{O}_{8+d}$  system, which is structurally and electronically very similar to the 123's, except for a slightly larger (5%) interplanar spacing. Interestingly, the superconductivity in this system is not quenched by a substitution of the rare earth with Pr, though it is quenched by substitution with Ce (the compound does not form for  $\text{R}=\text{Tb}$ ). We performed photoemission and resonant photoemission experiments on this compound to try to determine why the Pr doping has a different effect on the superconductivity in this class of compounds. We found Pr to have an

average valency of 3+, as in  $Y_{1-x}Pr_xBa_2Cu_3O_{6+d}$  and Pr metal. However, the extracted Pr 4f lineshape that we obtained from the resonant photoemission experiments showed evidence for weaker hybridization with the in-plane Cu and O states. In addition, the Pr 4f density of states peaked farther away from the Fermi level, implying that the states near the Fermi level, which are primarily responsible for the superconductivity, are probably least affected. This reduced hybridization can be explained by the increased interplanar spacing, and is consistent with the proposed mechanism for the quenching of the superconductivity in  $Y_{1-x}Pr_xBa_2Cu_3O_{6+d}$  but not in  $Pb_2Sr_2PrCu_3O_{8+d}$ .

#### *Angle-Resolved Photoemission studies of NiO and CoO*

The electronic structure of high  $T_c$  cuprate superconductors is related to that of transition metal compounds, which has been an interesting and controversial subject for a long time. Because of their simpler structures, transition metal mono-oxides like NiO and CoO provide opportunities for studies of the effects of the translational symmetry on the electronic structure of highly correlated materials such as the cuprate superconductors. We have performed angle-resolved photoemission studies of NiO and CoO. The results show that lattice effects are important to the electronic structure of NiO and CoO. For the oxygen derived bands, the magnitudes of the energy dispersions agree with band calculations. For the Ni 3d and Co 3d derived bands, however, our results do not agree with the band calculations.

#### *Monoclinic BaBiO<sub>3</sub>*

$Ba_{1-x}K_xBiO_3$  is the only known compound without Cu which has a transition temperature around 30 K or higher. The parent compound  $BaBiO_3$  of the superconductor  $Ba_{1-x}K_xBiO_3$  is interesting in that it has an odd number of electrons in the unit cell and hence is expected to be a metal; yet, experimentally it is found to be a semiconductor. An explanation invoking valence disproportionation between the two Bi ions to give  $Bi^{III}$  and  $Bi^V$  has been proposed to account for this discrepancy. We

have performed a resonant photoemission study of the monoclinic BaBiO<sub>3</sub>. The lineshapes of the valence band photoemission spectra and the Ba contributions to the valence band are very similar to the lineshapes of the total density of states and Ba partial density of states, respectively. The Bi 5d core levels show only one peak for each spin-orbit component. An oxygen resonance is observed, demonstrating the existence of empty O 2p states. These results support a more covalent rather than a simple ionic picture for the electronic states of BaBiO<sub>3</sub>. Our data suggest that the charge fluctuation between the unequal Bi sites is small. We also discuss the origin of the semiconducting character of the "valence disordered" BaBiO<sub>3</sub>.

#### *Electronic Structure of Copper Oxide Superconducting Thin Films*

Many different methods have been used to synthesize high T<sub>c</sub> thin films. We have performed photoemission studies to investigate the chemistry and the growth mechanism of the thin films during annealing under different conditions. We found the ionization state of Cu to strongly depend on its crystal environment and its oxidation process seems not to be reversible in a simple way. On the other hand, the ionization of Ba seems very inert, and Ba is stable in its doubly ionized state.



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Interaction of overlayers of Al and Rb with single crystalline surfaces of  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$   
Appl. Phys. Lett., submitted

P.A.P. Lindberg, Z.-X. Shen, D.S. Dessau, B.O. Wells, A. Borg, D.B. Mitzi, I. Lindau, W.E. Spicer, and A. Kapitulnik

Electronic structure of single crystalline  $\text{Bi}_2(\text{Sr,Ca,La})_3\text{Cu}_2\text{O}_8$

Physica C, submitted as contribution to the  $\text{M}^2$ -HTSC II Conference at Stanford University, July 1989

D.S. Dessau, Z.-X. Shen, P.A.P. Lindberg, B.O. Wells, A. Borg, I. Lindau, W.E. Spicer, J. Waszczak, and L.F. Schneemeyer

The electronic structure of  $\text{Pb}_2\text{Sr}_2\text{PrCu}_3\text{O}_8$  as studied by resonant photoemission spectroscopy

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Angle-resolved photoemission study of  $\text{NiO}$  and  $\text{CoO}$

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